MTO Reference Materials for Control, Calibration and Development of Soil and Aggregate Test Methods

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Abstract

The Ministry of Transportation of Ontario (MTO) has been actively involved in developing and establishing reference materials for soil and aggregate test method development, as well as test method control and calibration for over 35 years. The primary function of these materials is test method control and calibration for the critical purpose of ensuring the highest quality testing in support of durable Ontario infrastructure. Many of MTO's reference materials have been cited and used in test methods and publications by ASTM, CSA, RILEM, and numerous other internationally recognized research publications over the years. Many applied, practical and experimental research projects that are engaged in test method development and innovative technologies development are requestors of MTO's reference materials. These projects are normally in need of control materials with established or "known" parameters and/or established field performance to use in support of their research programs. MTO has historically both directly and indirectly greatly benefitted from this external research as a result of the practice of providing control materials.

This paper provides an overview of MTO's reference material program including its development and evolution. MTO's new reference material code system is presented, as well as the procedure for requesting materials and MTO's policies for making materials available to requestors.

A summary of the different reference materials currently available and their intended usage, such as which materials are used for control and calibration for which test methods, will be provided. There will be mention of the historical reference materials, that although may be commonly recognized, are now out of stock and no longer available, e.g., the "Brechin Aggregate". The newer materials that have superseded and replaced these historic ones are detailed, e.g., "Drain Brothers Stoney Lake". Currently there are ten to twelve different reference materials that are either actively in use and/or are in the process of being established for future use. Commonly known names for some of these materials include Spratt #3, Pittsburg, Sudbury Gravel, Dresden Clay, etc.

Reference material summaries include each material's physical and engineering property test ranges as well as descriptions of each material's field performance and geological environment of origin, where available. The chemistry and petrography for each is also included where available.

The reference material program demonstrates MTO's leadership and ongoing commitment to promoting quality testing of soils and aggregates for more durable infrastructure. MTO's reference materials have historically been used for and will continue to provide a solid foundation for test method development and research into the development of new and innovative technologies in the soils and aggregates field.

Introduction

Laboratory testing is a cost effective and essential practice to ensure quality materials are used in construction of our infrastructure. However, obtaining accurate and precise test measurements can be challenging due to issues with sampling, preparation, and variations in measurement systems, as well as technician skills. Variation in measurements between laboratories can result in disputes between parties on a construction contract. To resolve such disputes, a referee laboratory is typically invoked to perform testing and determine the final test results binding on both parties. Reference materials (RMs) provide a reliable means to minimize the variability of test measurements and therefore reduce the potential for such disputes.

Availability of suitable RMs are of key importance in aggregate and soil testing. Reference materials of known and stable parameters are of fundamental importance to both the pursuit of test method development and the overall quality of testing.

In general, reference materials (RMs) serve five main purposes in aggregate and soil testing:

- RMs provide a tool to demonstrate that testing is performed correctly, e.g., if the test results of the RM obtained are within the established ranges for the RM.
- RMs provide a tool to enable laboratories trouble shoot and/or adjust their procedures to obtain consistent test results, e.g., minimize variability of measurements both within a laboratory and between different laboratories.
- RMs provide a means of ensuring test results are traceable, e.g., through control charts.
- RMs provide a means of calibrating equipment and measurement systems.
- RMs of known parameters and of known field performance are necessary for future research and test method development.

Background & History

The Ministry of Transportation of Ontario (MTO) has been actively involved in developing and establishing reference materials for soil and aggregate test method development, as well as test method control and calibration for over 35 years. MTO's first foray into reference materials (RMs) was in the field of alkali-aggregate reactivity (AAR) during the mid-1980's. At that time, the available test methods for detecting potentially AAR aggregates were not very reliable and had poor correlations with the actual field performance of the aggregates. MTO's initial 3 RMs: Spratt, Sudbury and Pittsburg/Kingston were selected due to their established poor field performance of alkali-silica (ASR) and alkali-carbonate (ACR) reactivity. The main criteria for their selection was that the aggregate had been used in several concrete highway structures, and that these structures had deteriorated due to an alkali aggregate reaction (Rogers, 1988). At the time of acquisition, the primary purpose of curating these materials was to help promote development of new and better test methods for detecting potential AAR. It was reasoned that if the newly developed test methods could properly detect the materials with actual field performance problems, the test methods would also be successful in predicting the potential future field performance of materials for which there were no service records.

From the late 1980's through the 1990's, materials of known field performance were also used in the development of new aggregate durability tests such as the freeze-thaw test (MTO LS-614) and the micro-Deval tests for coarse and fine aggregates (MTO LS-618 and LS-619). It was also recognized that it would be necessary to develop and employ the use of RMs of appropriate characteristics with the test methods to ensure that laboratories were performing the testing correctly, consistently and to also provide a means of calibration and trouble shooting of procedures and equipment. Some of the commonly recognized names for these older and no longer available RMs include Brechin No. 2, Sutherland Sand and the Dresden Clay.

MTO Reference Material (MTO RM) Code System

In the spring of 2018, MTO began transitioning the naming of its reference materials (RM) over to a code system. The purpose of the code system is to allow the materials to be quickly categorized and classified according to their purpose. With the new naming convention also comes new statistical methods for establishing RM parameters, and new materials that supersede historical ones.

The code system therefore represents an effective ‘break’ with many of the historical methods that were used to establish the older MTO RMs. It also helps to reinforce that the parameters established for individual MTO RMs apply only to the individual stockpile that is being curated by MTO. In other words, MTO as the custodian of the RMs, only warrants the published parameters for an individual stockpile of material sourced by and in MTO custody. The same established parameters cannot be applied to new stockpiles, samples or other quantities of materials that may be derived from the same source. Even materials obtained from the same area within a source may not conform to the same parameters established for individual MTO RMs. Therefore, the materials’ characterization and established statistical parameters for each MTO RM only apply to each individual stockpile of material. When an RM is depleted, new parameters need to be established for any replacement material considered.

The code system also allows some of the older RMs that remain active to be refreshed using new data from current test methods and using new statistical methods, e.g., MTO RM ACR CA1. The code system also has the benefit of anonymizing the sources, particularly for newly acquired materials, thus protecting the identity of aggregate producers who have graciously provided their materials but prefer to be anonymous.

RM Code Breakdown and Categories

In the breakdown of the simple code system, MTO stands for “Ministry of Transportation, Ontario”, and RM stands for “Reference Material”. MTO RMs are further categorized by material type and/or intended purpose into three different categories: MTO RM AAR, MTO RM AGG and MTO RM Soil (Table 1).

Table 1. Categories of MTO Reference Materials.

MTO RM Category	Intended Purpose or Usage
MTO RM AAR	Reference aggregates intended for use with **alkali-aggregate reaction (AAR)** testing of aggregates. Test method control, test method calibration, test method development and research.
MTO RM AGG	Reference aggregates for general physical and engineering property testing of coarse and fine **aggregates**. Test method control and calibration.
MTO RM SOIL	Reference clays for **soil** testing

The acronyms CA and FA in the code system indicate whether the RM is a coarse or fine aggregate respectively. RMs for use with AAR testing also include the acronyms ASR, ACR and NRX to indicate if the RM is alkali-silica reactive (ASR), alkali-carbonate reactive (ACR) or non-reactive (NRX). The RMs for soil testing are simply referred to as Clays. The numbering of materials within each category and type is normally sequential and represents the succession and/or acquisition of new materials with time as the older RMs are depleted, become out of stock and are superseded.

The historic, currently active and pending release RMs within each category are summarized in Tables 2 through 4. The historical, out of stock and depleted materials were also included in the transition to the code system for the purpose of consistency and to show the progression, making users aware that there may have been a number of materials that preceded the one currently available for the intended use.

Table 2. MTO reference aggregates for alkali-aggregate reaction (AAR) testing and AAR test method development and research (MTO RM AAR).

MTO RM AAR Materials	Material Name or Source	Status
MTO RM ASR CA1	Spratt Quarry	Out of stock/Superseded
MTO RM ASR CA2	Spratt Quarry No. 2	Out of stock/Superseded
MTO RM ASR CA3	Spratt Quarry No. 3	Restricted availability

MTO RM ASR CA4	Sudbury Gravel	Restricted availability
MTO RM ASR CA5	Sudbury Gravel No. 2	Pending
MTO RM ASR CA6	Anonymous/Bobcaygeon Formation	Pending
MTO RM ACR CA1	Pittsburg Quarry	Active
MTO RM NRX CA1	Anonymous/Amabel Formation	Active
MTO RM NRX FA1	Masham Sand	Out of stock
MTO RM NRX FA2	Anonymous/Amabel Formation	Active

Table 3. MTO reference aggregates for general physical and engineering property testing of coarse and fine aggregates (MTO RM CA/FA).

MTO RM AGG Materials	Material Name or Source	Status
MTO RM CA1	Brechin Quarry No. 2	Out of stock/Superseded
MTO RM CA2	Drain Brothers Stoney Lake	Active
MTO RM FA1	James Dick Sand	Out of stock/Superseded
MTO RM FA2	Sutherland Sand	Out of stock/Superseded
MTO RM FA3	Anonymous	Active
MTO RM FA4	Stouffville Sand	Restricted availability

Table 4. MTO reference clays for soil testing (MTO RM Clay).

MTO RM Clay Materials	Material Name or Source	Details
MTO RM Clay1	Walker Clay	Out of stock/Discontinued
MTO RM Clay2	Dresden Clay	Active
MTO RM Clay3	Waterford Clay	Active
MTO RM Clay4	Heidelburg Clay	Active

Methodology for Establishment of RMs

The data used to establish the test method parameters and acceptable ranges for each MTO RM AGG and MTO RM SOIL material is normally collected through the annual Aggregate and Soil Proficiency Sample Testing Program (SASTP) that is conducted by the Soils and Aggregates Section of the Engineering Materials Office of the MTO.

It is recommended that testing of two duplicate samples by more than 15 laboratories will provide statistically adequate information to determine a meaningful estimate of single-operator precision (ASTM C802-14). For estimate of multi-laboratory variations, fewer than 10 laboratories is not desired. Currently, over two hundred private and public sector laboratories in the province of Ontario participate in this annual program. The program involves more than twenty tests conducted on duplicate samples of aggregate and soil materials from various sources in Ontario. The number of participating laboratories for the individual tests varies from year to year, as each laboratory may not participate in every test that is allotted to a particular material. Each participating laboratory is supplied with a pair of nearly identical samples so that single laboratory repeatability can be assessed. The data and methods used to establish the parameters and acceptable ranges for the different RMs vary depending on the test methods as well as past and present practices.

Many of the AAR RMs, prior to the more recent MTO RM ASR CA5 and MTO RM ASR CA6, were generally established through interlaboratory studies completed either external to MTO and/or in partnership with other associations, agencies or research entities. Often these older interlaboratory studies relied upon a single sample tested per laboratory, i.e., not a pair. Since 2015, MTO has been applying the same practice of supplying duplicate samples for the AAR testing and establishment of the AAR RMs. The AAR testing has also been included routinely as part of the annual SASTP. This has overall resulted in better oversight, improved consistency and more robust parameters established for MTO's RM's.

Outlying data may introduce bias into calculations of variability and precision of normal test methods. The probability of such outliers occurring by chance is statistically very small. A technique designed by AMRL was used to remove outlying data from the MTO proficiency test results and to analyze the remaining core data (Holsinger et al., 2005). For test methods generating a single determination as a test result, single operator (repeatability) and multi-laboratory (reproducibility) precision estimates are established through the AMRL analysis. For the other test methods requiring calculation of 2 or more replicate determinations from the same batch to generate the final test result, a procedure as defined in ASTM C802-14 was used to analyze the core data to establish single operator precision for variability within batch single-operator, multi-batch precision and multi-laboratory precision. Further details of the analytical process are included in Engineering Materials Office Report 211 (EMO-211).

Each material will be discussed in the following sections. Further details of each reference material including the data used to establish the newer and pending RMs are included in EMO-211.

RM Descriptions

MTO RM AAR

MTO RM ASR CA1 (Spratt No. 1)

MTO RM ASR CA1 or Spratt No. 1 was the first of three 100 tonne stockpiles of AAR aggregates established by MTO in 1986 (Fournier et al., 2012). The material was acquired due to its service records including demonstrated problems with ASR when used in concrete. This ASR coarse aggregate RM was derived from and named after the Spratt Quarry near Ottawa, Ontario. Spratt No. 1 consisted of a siliceous limestone derived from the upper Bobcaygeon Formation.

Aggregate test data, petrographic composition and chemical composition of Spratt No. 1 is summarized in Tables 5 through 7. Spratt No. 1 was provided free of charge by MTO to encourage AAR research and test method development until it was exhausted in 1991 for the construction of an outdoor exposure site (Fournier et al., 2012; MacDonald and Rogers, 2012, Hooton et al., 2013). This material was superseded by the Spratt No. 2 in 1992 (see MTO RM ASR CA2).

Table 5. Test data for Spratt No. 1. Result is an average of n = 4 samples, except insoluble residue where n = 1 (Rogers, 1988, and Rogers & MacDonald, 2012).

MTO LS	Coarse Aggregate Test	Result
LS-601	Wash Pass 75 µm	0.32%
LS-603	LA Abrasion and Impact	18.3%
LS-604	Relative Density	2.682
	Absorption	0.46%
LS-606	Magnesium Sulphate Soundness	1%
LS-613	Insoluble Residue (IR_{total}) n = 1	10.0%
LS-609	Petrographic Number	111

Table 6. Petrographic composition of Spratt No. 1. Result is an average of n = 4 samples from different areas of the Spratt No. 1 stockpile (Rogers, 1988).

Petrographic Composition	Result
Good Limestone	96.3%
Slightly Shaley Limestone	2.3%
Cherty Limestone	0.5%
Shaley Limestone	0.8%
Shale	0.1%
Total	100%

Table 7. Chemical analysis of Spratt No. 1 and of the insoluble residue (IR_{Total}) of Spratt No. 1, n = 1 sample (Rogers, 1988).

Element, %	Spratt No. 1 Aggregate	Spratt No. 1 IR (10.0%)
SiO_2	8.70	86.92
TiO_2	0.04	0.21
Al_2O_3	0.59	4.24
Fe_2O_3	0.58	1.28
MgO	1.67	0.78
CaO	48.47	0.26
Na_2O	0.04	0.08
K_2O	0.08	0.78
P_2O_5	0.29	0.45
LOI	39.55	4.02
Total	100.14	100.18
S	0.13	1.16

There are no established parameters for this material for the accelerated mortar bar (AMBT) and concrete prism expansion (CPT) tests as this RM was depleted while these test methods were still in the developmental stages.

MTO RM ASR CA2 (Spratt No. 2)

A second 100 tonne stockpile from the same bench in the Spratt Quarry as Spratt No. 1 was acquired by MTO and established in 1992. After qualification of Spratt No. 2 through interlaboratory studies (Fournier and Malhotra, 1996; Rogers, 1996; Rogers, Boothe and Jiang, 1996, and Rogers, 1999); this material (Spratt No. 2) officially superseded the depleted Spratt No.1.

A summary of the ranges for AMBT and CPT at 38°C for the Spratt No. 2 is summarized in Table 8.

Table 8. Established AAR test parameters for the Spratt No. 2 (Rogers, 1999; and Fournier and Malhotra, 1996). Data ranges are based on single samples tested in two interlaboratory studies with n = 41 AMBT and n = 27 CPT participants respectively.

AAR Test	Expansion Range
Accelerated Mortar Bar Test (14 days)	0.30 – 0.55%
Accelerated Mortar Bar Test (28 days)	0.47 – 0.98%
Concrete Prism Expansion Test (1 year)	0.12 – 0.23%
Concrete Prism Expansion Test (2 years)	0.15 – 0.29%

Other than the AAR testing completed during the interlaboratory studies to establish Spratt No. 2, some other physical and engineering property testing was completed (Table 9). It was assumed that because the material was derived from the same bench of the same quarry, other test data parameters, e.g., LA abrasion and impact, magnesium sulphate soundness and petrographic composition would remain stable and consistent with that of Spratt No. 1 (Table 5).

However, it is interesting to note the increase in insoluble residue (IR) content in Spratt No. 2 as compared with Spratt No. 1 (Tables 5 and 9 respectively). Additionally, a comparison of the chemical analyses of Spratt No. 1 and Spratt No. 2 (see second columns of Tables 7 and 10 respectively) also shows relative increases in the amount of SiO_2 major oxide. This major oxide generally occurs within the insoluble mineral phases in the aggregate, in particular the silica present as chert, chalcedony and possibly quartz that forms the alkali-silica reactive component of Spratt No. 1 and Spratt No. 2. The relative increases in both IR and SiO_2 in Spratt No. 2 as compared with Spratt No. 1 also suggest the former may have been more reactive and therefore more expansive than the latter.

Table 9. Test data for the Spratt No. 2 (Rogers & MacDonald, 2012; MTO unpublished internal files).

MTO LS	Coarse Aggregate Test	Result
LS-603	LA Abrasion and Impact	19.0%
LS-604	Relative Density	2.712
	Absorption	0.72%
LS-613	Insoluble Residue (IR_{total})	11.65%

The Spratt No. 2 stockpile was largely depleted between 2006 and 2009. During that time, supply of the material was mainly restricted to supporting test method control and calibration within Ontario (no research). The restriction to supply the materials for research purposes was lifted once Spratt No. 2 was officially superseded by the Spratt No. 3 in 2012 with the publication of Fournier et al. (2012) (see MTO RM ASR CA3).

Table 10. Chemical analysis of Spratt No. 2 and of the insoluble residue (IR_{Total}) of Spratt No. 2, n = 1 sample (EMO-211).

Element, %	Spratt No. 2 Aggregate	Spratt No. 2 IR (11.65%)
SiO_2	10.56	81.22
TiO_2	0.02	0.23
Al_2O_3	0.52	4.34
Fe_2O_3	0.26	1.33
MnO	0.03	0.01
MgO	1.69	0.69
CaO	47.798	1.2
Na_2O	0.03	<0.01
K_2O	0.07	0.9
P_2O_5	0.172	0.04
LOI	38.95	8.55
Total	100.1	98.47
S	0.073	1.38

MTO RM ASR CA3 (Spratt No. 3)

A third 100 tonne stockpile from the Spratt Quarry was acquired by MTO in 2006 and formally established through publication of an international interlaboratory study in 2012 (Fournier et al., 2012). Upon publication of the 2012 paper, Spratt No. 3 officially superseded Spratt No. 2.

A summary of the ranges established for the AMBT and CPT at 38°C and 60°C for Spratt No. 3 is summarized in Table 11.

Table 11. Established AAR test parameters for the Spratt No. 3 (Fournier et al., 2012). Data ranges are based on single samples tested with n = 49 AMBT at 14 days, n = 50 AMBT at 28 days, 37 CPT at 38°C and 21 CPT at 60°C.

AAR Test	Expansion Range
Accelerated Mortar Bar Test (14 days)	0.28 – 0.49%
Accelerated Mortar Bar Test (28 days)	0.45 – 0.73%
Concrete Prism Expansion Test 38°C (1 year)	0.1 – 0.29%
Concrete Prism Expansion Test 60°C (6 months)	0.08 – 0.23%

Other historical physical and engineering property testing for Spratt No. 3 is included in Table 12 and EMO-211. Similar to Spratt No. 2, it was assumed that because the material was derived from the same area of the quarry as previous Spratt stockpiles, that other test data parameters, e.g., petrographic composition would remain stable to that of Spratt No. 1 (Table 5).

Table 12. Test data for Spratt No. 3 (MTO UIF; Rogers & MacDonald, 2012; EMO-211).

MTO LS	Coarse Aggregate Test	Result
LS-603	LA Abrasion and Impact	19.0%
LS-604	Relative Density	2.712%
	Absorption	0.72%
LS-613	Insoluble Residue (IR_{total})	9.95 – 10.15%

An average of 6 chemical analyses of Spratt No. 3 are provided in Table 13. In general, the results show a marked decrease in both the amount of insoluble residue (Tables 12 and 13) and in the SiO_2 contents as compared with that of the Spratt No. 1 (Tables 5 and 7) and Spratt No. 2 (Tables 9 and 10). This suggests that the reactive component of the Spratt No. 3, i.e., silica in the form of chert, chalcedony and possibly poorly crystalline quartz, may have been present in lesser amounts as compared with the first two stockpiles. This may have resulted in slightly lower reactivity and therefore less expansivity in the AMBT and CPT as compared with Spratt No. 1 and Spratt No. 2. This is consistent with the slightly lower established expansion for Spratt No. 3 (Table 11) as compared with that for Spratt No. 2 (Table 8). Reports from users have also corroborated less expansion with Spratt No. 3 versus Spratt No. 2 in practice.

Table 13. Average chemical analysis of Spratt No. 3, (n = 6), and of the insoluble residue (IR_{Total}) of Spratt No. 3, n = 1 analysis (EMO-211). Bdl = below detection limit.

Element, %	Range	Spratt No. 3 Aggregate	Spratt No. 3 IR (10.05%)
SiO_2	1.43	7.86	84.04
TiO_2	0.03	0.03	0.17
Al_2O_3	0.13	0.47	3.95
Fe_2O_3	0.05	0.26	1.63
MnO	0.01	0.04	0.01

Element, %	Range	Spratt No. 3 Aggregate	Spratt No. 3 IR (10.05%)
MgO	0.36	1.75	0.65
CaO	1.68	48.94	1.01
Na_2O	bdl	<0.01/<0.02	<0.01
K_2O	0.04	0.08	0.88
P_2O_5	0.02	0.18	0.04
LOI	2.78	41.21	7.48
Total	1.85	100.78	99.85
S	0.04	0.07	1.18

MTO RM ASR CA4 (Sudbury Gravel)

MTO RM ASR CA4 or Sudbury Gravel or simply Sudbury Aggregate is one of the first three original 100 tonne AAR aggregate stockpiles acquired by MTO in 1986. The material was acquired due to its service records including demonstrated problems with ASR when used in concrete, e.g., Dolar-Mantuani, 1969 and Gratten-Bellew, 1978. Magni et al. (1987) effectively documented the poor field performance of the local gravels when used in concrete by reporting the characteristic symptoms of ASR in at least 26 concrete structures and other non-structural concretes in the Sudbury area that had developed expansion and cracking due to ASR. Jones et al., 1990 also documented the problem as well as presenting one of the solutions MTO had implemented for a time, i.e., through limiting the amount of reactive components allowable through petrographic analysis.

The aggregate consists of a partly crushed gravel derived from a pit source near the Sudbury Airport that is located within a kame/delta deposit (Rogers, 1988, and Rogers and MacDonald 2012). In general, the material is composed of hard, durable siliceous rock types that are derived from the underlying Canadian Shield bedrock. Lithologies present include a mixture of sedimentary rocks of Proterozoic age, as well as other Precambrian high grade metamorphic and igneous rocks.

A summary of the established data for the Sudbury Gravel in the CPT at 38°C (CPT) is summarized in Table 14.

Table 14. Established CPT parameters for the Sudbury Gravel (Fournier and Malhotra, 1996). Data ranges are based on single samples tested with n = 27 CPT at 38°C.

Concrete Prism Expansion Test at 38°C	Expansion
Average 52-week expansion (1 year)	0.093%
Standard deviation	0.037%
Coefficient of variation	39.6

Aggregate test data and petrographic composition of the Sudbury Gravel is summarized in Tables 15 and 16. A breakdown of the petrographic composition is provided in Table 16. The reactive components, include argillite, greywacke, sandstone, arkosic sandstone, and quartzite which together compose approximately 75% of the material (Table 16).

Table 15. Test data for Sudbury Gravel, n = 3, (Rogers, 1988, and Rogers & MacDonald, 2012).

MTO LS	Coarse Aggregate Test	Result
LS-601	Wash Pass 75 µm	1.51%
LS-603	LA Abrasion and Impact	13%
LS-604	Relative Density	2.710
	Absorption	0.46%

MTO LS	Coarse Aggregate Test	Result
LS-606	Magnesium Sulphate Soundness	1%
LS-609	Petrographic Number	117

Table 16. Historical petrographic composition of the Sudbury Gravel, n = 3 samples (Rogers, 1988; and Rogers & MacDonald, 2012).

Petrographic Composition	Result
Sandstone and Arkosic Sandstone	31.5%
Argillite and Greywacke	32.3%
Quartzite and Quartz	11.1%
Granite, Diabase, Gneiss, etc.	25.1%
Total	100%

The Sudbury Gravel was provided free of charge by MTO to encourage AAR research and test method development until it was recently exhausted. This material is intended to be superseded by the Sudbury Gravel No. 2 or MTO RM ASR CA5 which is currently pending official release (see below).

MTO RM ASR CA5 (Sudbury Gravel No. 2)

MTO RM ASR CA5 is derived from a gravel deposit located northeast of Sudbury, Ontario near the Sudbury airport. The source location is either at or in close proximity to the original source for MTO RM ASR CA4. MTO RM ASR CA5 is intended to supersede the depleted MTO RM ASR CA4. It will therefore continue to fulfill a role as one of the three main alkali-aggregate reaction (AAR) reference materials supplied by MTO to develop new test methods, mitigation strategies and for test method control and calibration.

MTO RM ASR CA5 consists of predominantly hard, siliceous gravel derived mainly from the Proterozoic and Archean bedrock formations (Canadian Shield) that underlie and surround the gravel deposits in the Sudbury region. Composition is comparable to MTO RM ASR CA4 (see below).

Prior to MTO acquiring the MTO RM ASR CA5 stockpile, its petrographic composition and AMBT results were compared to those of MTO RM ASR CA4. This was completed to establish that the two materials were compositionally comparable and logically that the properties of the superseding material would likewise be reasonably consistent with the original (Table 17).

Table 17. Preliminary comparative test data between the Sudbury Gravel (MTO RM ASR CA4) and MTO RM CA5 to assess suitability prior to acquisition of new material.

Test	MTO RM ASR CA4	MTO RM ASR CA5
Accelerated Mortar Bar Test (14 days)	0.257%	0.268%
Petrographic Number	101	101

Table 18. Comparison of petrographic compositions of MTO RM ASR CA4 and MTO RM CA5 to assess suitability prior to acquisition of the MTO RM CA5 stockpile.

Petrographic Composition	MTO RM ASR CA4	MTO RM ASR CA5
Sandstone and Arkosic Sandstone	40.86%	46.38%
Greywacke – Argillite	29.26%	24.81%
Quartzite	4.69%	5.61%
Gabbro – Diorite	15.07%	11.60%
Granite	4.63%	4.68%

Mafic Metavolcanic and/or Trap	5.08%	6.42%
Felsic Metavolcanic	0.37%	0.21%
Other	0.04%	0.29%
Total	100.0%	100.0%

MTO RM ASR CA5 has been included in several SASTPs since 2017. Data from these proficiency programs are currently being used to establish this new material. Detailed raw data and the statistical methods used to establish this new stockpile can be found in EMO-211.

MTO RM ASR CA6 (Anonymous - Bobcaygeon Formation)

MTO RM ASR CA6 consists of a quarried carbonate aggregate that is derived from the Ordovician-aged Bobcaygeon Formation, in Eastern Ontario (Derry Michner Booth and Wahl and OGS, 1989; Armstrong & Dodge, 2007). MTO RM ASR CA6 is intended to supersede and replace MTO RM ASR CA3/Spratt No. 3 which is currently under restricted availability due to the limited quantities remaining. Prospecting for a new AAR RM source in the same geological formation as, and with comparable characteristics to Spratt No. 3 was undertaken by MTO between 2018 and 2020. Screening of prospective sources typically included AMB testing to assess the level of reactivity, and IR testing to assess the general composition.

Once a potentially suitable source was identified, further AMBT testing was conducted on material from the prospective source and on the Spratt No. 3, concurrently at two different laboratories (Table 19). IR testing of the two materials was also conducted (to provide information regarding the composition) but only by the second laboratory (Table 19). IR and AMBT results for the two materials were observed to compare well (Table 19), suggesting possibly similar behavior in terms of composition and level of reactivity, therefore supporting the decision to move forward with this source as a potential replacement for Spratt No. 3. The aggregate producer has chosen to be anonymous, hence the materials can not be identified by the source name or its specific location. The material shall be referred to by its code name: MTO RM ASR CA6. Further details are provided below to help characterize this new material.

Table 19. MTO RM ASR CA6 preliminary comparative test data.

Test	MTO RM ASR CA3 Spratt No. 3	MTO RM ASR CA6 Anonymous
Accelerated Mortar Bar Test (14 days) (Lab 1)	0.429%	0.445%
Accelerated Mortar Bar Test (14 days) (Lab 2)	0.420%	0.413%
Insoluble Residue (IR_{total}) (Lab 2)	9.91%	10.29%

The primary intended application of MTO RM ASR CA6 is as an alkali-silica reactive control material for use in test method LS-635, Length Change due to Alkali-Aggregate Reaction in Concrete Prisms at 38°C, and in test method LS-620, Accelerated Detection of Potentially Deleterious Alkali-Silica Reactive Aggregate by Expansion of Mortar Bars.

MTO RM ASR CA6 was included in the SASTPs for 2021 and 2022. Data from these proficiency programs are currently being used to establish this new material. Detailed raw data and the statistical methods used to establish this new stockpile can be found in EMO-211. A summary of the aggregate test results from the 2021 SASTP for MTO RM ASR CA6 is included in Table 20.

Table 20. Summary of aggregate test data for MTO RM ASR CA6 (EMO-211 and EMO-213 and EMO-214).

MTO LS	Coarse Aggregate Test	n	Multi-laboratory Variation	Mean
LS-601	Wash Pass 75 µm	199	Standard deviation = 0.27	1.44%

LS-603	LA Abrasion and Impact	11	Coefficient of variation = 3.9%	17.6%
LS-604	Relative Density	88	Standard deviation = 0.005	2.677
	Absorption	89	Standard deviation = 0.057	0.44%
LS-606	Magnesium Sulphate Soundness	38	Coefficient of variation = 26.5%	3.9%
LS-613	Insoluble Residue, Total	11	Standard deviation = 0.8	12.9%
LS-614	Unconfined Freeze-Thaw	52	Coefficient of variation = 22.5%	5.9%
LS-618	Micro-Deval Abrasion	75	Coefficient of variation = 4.9%	8.0%
LS-620	Accelerated Mortar Bar	28	Coefficient of variation = 8.5%	0.484%
LS-635	38°C Concrete Prism Test	14	Coefficient of variation = 16.0%	0.175%

The test data in Table 20 shows that the MTO RM ASR CA6 material may be capable of meeting many of the requirements of aggregates for use in MTO structural concrete, with the notable exception of the AAR test results. The range in LS-620, AMBT, results of 0.401 to 0.565% expansion at 14 days (Table 20) clearly indicates that MTO RM ASR CA6 can be classified as highly to extremely alkali-silica reactive (ASR) according to Table 2 of CSA A23.2-27A (CSA, 2019).

Compositionally, MTO RM ASR CA6 consists of a medium to dark grey brown and medium grey limestone (visual and field examination). Trace amounts of shale in the form of laminations occur locally as do minor amounts of light grey to dark coloured chert. Trace amounts of pyrite are also visible locally and may typically be associated with the presence of chert. A summary of the petrographic composition of MTO RM ASR CA6 based on examination of 20 samples is provided in Table 21.

Additional information with regards to composition is further supplied by insoluble residue results for 20 samples, each representing one of the truckloads of MTO RM ASR CA6 delivered to form the stockpile (Table 22). The IR results occur within a very narrow range of 11.59 – 12.91% indicating a very stable, homogeneous material. Petrographic number (PN) results for the same 20 samples also show remarkable consistency with a range of 101 to 104 (Table 22).

Table 21. Petrographic composition of MTO RM ASR CA6 (EMO-204). Results are an average of n = 20

Petrographic Composition	Average
Good Limestone	97.6%
Slightly Shaley Limestone	0.4%
Cherty Limestone	2.0%
Shaley Limestone	0%
Shale	0%
Total	100%

Table 22. Petrographic number (PN) and IR test results of MTO RM ASR CA6 (EMO-204). n = 20

Test	Range	Average
Petrographic Number (PN)	4 (101 – 106)	104
Insoluble Residue (IR_{total})	1.32% (11.59 – 12.91%)	12.27%

Geochemical analyses for 20 samples of MTO RM ASR CA6, each representing one of the truckloads of MTO RM ASR CA6 delivered to form the stockpile (Table 23) confirms that the material can be properly classified as a siliceous limestone. Ranges of the major oxides show that the material meets the criteria for classification primarily as limestone under the classification of Hewitt (1960) with total MgO contents ranging from 1.58 to 1.69%, CaO contents from 46.750 to 47.302%, and CaO:MgO ratios that have a relatively narrow range of 27.74 to 29.73% (EMO-211 and Table 23). As there are impurities present in the MTO RM ASR CA6 limestone aggregate, mainly in the form of chert as indicated by the approximately 11 – 12% SiO_2 and minor argillaceous content, the CaO:MgO ratio is used for the overall reference aggregate classification as limestone or siliceous limestone.

Table 23. Average chemical analysis of MTO RM ASR CA6, (EMO-204, EMO-211, EMO-213).

Chemical Analysis of MTO RM ASR CA6, n = 20		
Element, %	**Range**	**Average**
SiO_2	1.03%	11.40%
TiO_2	0.00%	0.01%
Al_2O_3	0.04%	0.43%
Fe_2O_3	0.02%	0.23%
MnO	0.003%	0.030%
MgO	0.11%	1.62%
CaO	0.552%	47.067%
Na_2O	0.01%	0.03%
K_2O	0.02%	0.05%
P_2O_5	0.006%	0.138%
LOI	0.44%	38.62%
Total	0.34%	99.63%
S	0.018%	0.046%

Table 24. Average chemical analysis of the insoluble residues of MTO RM ASR CA6, (EMO-204, EMO-211 and EMO-213).

Chemical Analysis of MTO RM ASR CA6 Insoluble Residues, n = 20		
Element, %	**Range**	**Average**
SiO_2	8.14%	88.87%
TiO_2	0.09%	0.12%
Al_2O_3	0.77%	1.73%
Fe_2O_3	0.50%	1.11%
MnO	0.004%	0.004%
MgO	0.46%	0.32%
CaO	1.715%	0.940%
Na_2O	0.15%	0.18%
K_2O	0.14%	0.35%
P_2O_5	0.008%	0.015%
LOI	5.84%	5.89%
Total	1.44%	99.53%
S	0.208%	0.832%

The very narrow ranges in major element oxide contents (Table 23), insoluble residue test results (Table 22) and major element oxide contents for the insoluble residues (Table 24) of twenty properly prepared samples from MTO's MTO RM ASR CA6 stockpile effectively demonstrates this reference aggregate to be an extremely consistent and relatively homogeneous material with respect to composition.

It is expected that once formally established, that MTO RM ASR CA6 will fulfill a prominent role as one of the three main alkali-aggregate reaction (AAR) reference materials supplied by MTO for development, control and calibration of test methods and research into mitigation strategies.

MTO RM ACR CA1 (Pittsburg)

MTO RM ACR CA1 is supplied as an alkali carbonate reactive (ACR) material for research and test development purposes. MTO RM ACR CA1 was also previously known or referred to as the "Pittsburg aggregate" (Rogers & MacDonald, 2012) and also as the "Kingston aggregate". This material is one of the first three original 100 tonne AAR aggregate stockpiles acquired by MTO in 1986.

Use of this coarse aggregate in structural concrete has been demonstrated to cause severe expansion and cracking within 2 to 5 years post construction (Rogers, 1985; Rogers, 1986, and Rogers, 1988). Historical expansion data reported for the 1-year CPT at 38°C ranges from 0.212 to 0.446% (Rogers & MacDonald, 2012; Rogers, 1988) (Table 25). It is important to note that this historical testing would have been completed during the 1980's and early 1990's while the CPT was still in the developmental stages.

Table 25. Historical CPT parameters for the Pittsburg Aggregate (Rogers, 1992). Data ranges are based on single samples tested with n = 16 laboratories.

Testing Conditions	Age (Days)	Range of Expansion	Mean Expansion, %	Standard Deviation	Coefficient of Variation
Box at 38°C	365	0.2120 - 0.4461	0.3069	0.07160	23.3%

Limited historical aggregate test data available for the Pittsburg aggregate are included in Table 26 to provide a general idea of the character of the material. Although the historical $MgSO_4$ loss of 1% suggests that the aggregate is not susceptible to damage from freezing and thawing, the Pittsburg aggregate is known to demonstrate poor field performance in terms of lack of frost durability (Rogers, 1983).

Table 26. Historical test data for the Pittsburg Aggregate (Rogers, 1988).

MTO LS	Coarse Aggregate Test	Result
LS-601	Wash Pass 75 µm	1.55%
LS-603	LA Abrasion and Impact	18%
LS-604	Relative Density	2.682
	Absorption	0.46%
LS-606	Magnesium Sulphate Soundness	1%
LS-609	Petrographic Number	127
LS-613	Insoluble Residue (IR_{total})	12.2%

Petrographically, the aggregate consists of a medium grey to greenish grey dolomitic limestone. A summary of the historical petrographic composition for the Pittsburg is included in Table 27.

Table 27. Historical petrographic composition of the Pittsburg Aggregate, n = 4 (Rogers, 1988).

Petrographic Composition	Result
Good Dolomitic Limestone	91.9%
Slightly Shaley Dolomitic Limestone	5.5%
Shaley Dolomitic Limestone	2.1%
Shale	0.5%
Total	100%

Geochemical analyses of MTO RM ACR CA1 (Table 28) confirm that the aggregate meets the criteria for classification as a dolomitic limestone under the classification of Hewitt (1960). Argillaceous content is predominantly reflected in the SiO_2 and Al_2O_3 contents (MacDonald, 2022 and EMO-211). Additionally, the total insoluble residue result of 12.2% for this reference aggregate (Table 26) further reflects the non-carbonate mineral content that is considered an impurity under Hewitt's (1960) classification.

Table 28. Average chemical analysis (XRF) of MTO RM ACR CA1/Pittsburg, (n = 5), and of the insoluble residue of Pittsburg, (n = 1) (EMO-204 and EMO-211).

Element, %	Range	Average	Analysis of Residue IR (12.2%)
SiO_2	0.64	6.83	61.25
TiO_2	0.03	0.10	0.73
Al_2O_3	0.31	1.86	16.2
Fe_2O_3	0.08	0.75	3.38
MnO	0.01	0.03	0.01
MgO	1.42	5.94	2.66
CaO	0.63	42.54	0.35
Na_2O	0.04	0.07	0.31
K_2O	0.19	0.64	6.21%
P_2O_5	0	0.02	0.02%
LOI	2.66	41.48	6.54%
Total	1.13	100.18	97.6%
S	0.06	0.06	0.77%

MTO RM ACR CA1 has been utilized in several SASTPs since 2019. This includes tests: LS-609, LS-613, LS-615, LS-616, LS-620 (Pair 2019 M1), and CSA A23.2-14A as part of the 2019 program, and LS-635 as part of the 2020 and 2022 programs. Results for these tests can be found in the SASTP reports, e.g., EMO-200, EMO-202, EMO-205, EMO-214 and EMO-215. It is also this data that will be used to establish new parameters for MTO RM ACR CA1. Further details including the raw data and explanations of the statistical methods used can be found in report EMO-211.

MTO RM NRX CA1 (Anonymous Source in Amabel Formation)

MTO RM NRX CA1 is a non-alkali-aggregate reactive (non-AAR) coarse aggregate RM for use with test method LS-635. LS-635 specifies the use of MTO reference aggregates including non-AAR fine and coarse reference aggregates for the evaluation of potential reactivity of coarse and fine aggregates respectively.

MTO RM NRX CA1 mainly consists of a grey to grey-blue to yellowish buff, coarse to fine crystalline reefal dolostone that is quarried from the Silurian-aged Amabel Formation, in the Niagara Escarpment of the Greater Golden Horseshoe area of Ontario. Aggregate mined from the Amabel Formation in this RM source area has commonly been utilized as a premium source of high-quality concrete aggregate in the Province of Ontario for over 50 years (Derry Michner Booth and Wahl and OGS, 1989).

MTO RM NRX CA1 has been included in in at least two SASTPs since 2017. Data from these proficiency programs are currently being used to establish this new material. Detailed raw data and the statistical methods used to establish this new stockpile can be found in EMO-203 and EMO-211.

MTO RM NRX FA2 (Anonymous Source in Amabel Formation)

MTO RM NRX FA2 has been included in several SASTPs since 2017. Data from these proficiency programs are currently being used to establish this new material. Detailed raw data and the statistical methods used to establish this new stockpile can be found in EMO-211.

MTO RM AGG

MTO RM CA1 (Brechin Quarry No. 2)

MTO RM CA1 or Brechin Quarry No. 2 or Brechin No. 2 Aggregate was one of the first officially recognized reference aggregates to be used primarily as a control and calibration material for many of the coarse aggregate durability and physical property tests including micro-Deval abrasion testing, i.e., MTO LS-618 (2004), CSA A23.2-29A (2004), ASTM D 6928-03 and AASHTO TP 58-03. The Brechin Aggregate was derived from a quarry operating in the Carden Plain area north of the Greater Toronto Area near Lake Simcoe. The material consists predominantly of a shaley limestone derived from the Bobcaygeon and possibly Verulam geological formations. Established parameters for the Brechin No. 2 Aggregate are included in Table 30.

Table 30. Established parameters for the Brechin Quarry No. 2, n = 35 and 86 laboratories.

Coarse Aggregate Test	Established Range	Average
Dry Relative Density	2.658 – 2.682	2.670
Absorption	0.55 – 0.81%	0.68%
Magnesium Sulphate Soundness	8.0 – 18.8%	13.2%
Unconfined Freeze-Thaw	10.2 – 20.9%	15.6%
Micro-Deval Abrasion	17.5 – 20.7%	19.1%

The Brechin No. 2 Aggregate was depleted and superseded by the Drain Brothers Stoney Lake Quarry Limestone in 2010/2011 (see MTO RM CA2 below).

MTO RM CA2 (Drain Brothers Stoney Lake Quarry Limestone)

MTO RM CA2 consists of a quarried carbonate aggregate that is derived from the Ordovician-aged Gull River Formation, near Havelock, Ontario. MTO RM CA2 is supplied as a control material for various aggregate tests including coarse aggregate micro-Deval (LS-618), magnesium sulphate soundness loss (LS-604), and unconfined freeze-thaw tests (LS-614), etc. (Table 31). MTO RM CA2 was also previously known as the Drain Brothers Stoney Lake Quarry Limestone (Vasavithasan & Senior, 2010). Aggregate test results for MTO RM CA2 are summarized in Table 31.

Table 31. Aggregate test data for MTO Reference Aggregate MTO RM CA2 (Vasavithasan and Senior, 2010).

MTO LS	Coarse Aggregate Test	Range	Mean
LS-603	LA Abrasion and Impact	23.2 – 28.8%	26.0%
LS-604	Relative Density	2.681 – 2.699	2.690
	Absorption	0.29 – 0.49%	0.39%
LS-606	Magnesium Sulphate Soundness	4.9 – 12.9%	8.9%
LS-614	Unconfined Freeze-Thaw	8.5 – 15.3%	11.9%
LS-618	Micro-deval Abrasion	11.4 – 14.8%	13.1%

Petrographically, MTO RM CA2 mainly consists of lithographic and locally stylolitic limestone, and with minor shale content and locally sparse fossil content. Chert and chert-bearing carbonate also forms a relatively minor component of the MTO RM CA2. Trace amounts of sandy carbonate may also be present locally. Small amounts of aggregate cross contamination may be present in the form of mafic volcanic (traprock) particles and to a much lesser extent granite particles that are almost exclusively present within the P9.5/R4.75 mm fraction (EMO-204 and EMO-211).

Geochemical analyses of MTO RM CA2 (Table 32), further confirm that this reference aggregate may be compositionally classified as a limestone according to Hewitt's (1960) classification. Total MgO contents for MTO RM CA2 range from 0.86 to 0.97% (Table 32), CaO contents range from 51.21 to 54.28%, and CaO:MgO ratios range from 52.79 to 61.25%. As there are impurities present in the MTO RM CA2 limestone aggregate, mainly in the form various argillaceous and chert content, the CaO:MgO ratio is used for the overall reference aggregate classification as limestone. Argillaceous content, e.g., shale laminations, is primarily reflected in the Al_2O_3 and SiO_2 contents that range from 0.74 to 1.17% and 3.87 to 5.25% respectively. The ranges in SiO_2 content are predominantly due to the chert content and trace amounts of arenaceous (sand) content. Inclusion of small amounts of mafic volcanic and possibly granite aggregate cross contamination, mainly in the P9.5/R4.75 fraction, may have also contributed to increased amounts of some of the major oxides, including SiO_2 and Al_2O_3 as compared to the limestone aggregate on its own.

Table 32. Average chemical analysis (XRF) of MTO RM CA2 (EMO-203 and EMO-211).

Chemical Analysis of MTO RM CA2, n = 12		
Element, %	**Range**	**Average**
SiO_2	1.38	4.43
TiO_2	0.04	0.06
Al_2O_3	0.34	1.00
Fe_2O_3	0.15	0.42
MnO	0.01	0.04
MgO	0.11	0.91
CaO	3.07	52.45
Na_2O	0.04	0.07
K_2O	0.12	0.22
P_2O_5	0.002	0.01
LOI	0.94	41.23
Total	1.97	100.75
S	0.035	0.056

Other MTO RMs

Descriptions, detailed data and the statistical methods used to establish the remaining MTO RMs, e.g., MTO RM NRX FA1, MTO RM FA2, MTO RM FA3, MTO RM FA4, MTO RM Clay2, MTO RM Clay3 and MTO RM Clay 4 can be found in EMO-211.

MTO Policy on Reference Material Availability

The primary purpose and justification for maintaining MTO RMs is for test method control and calibration to support MTO contract administration. By testing of RMs and producing results that are within the established range, RMs provide the means to validate test method procedures and reliability of the test results. RMs also ensure that high quality reliable data is used to decide the acceptance of materials for their intended purpose.

Availability and supply of RMs to laboratories that conduct quality testing for assessment of soil and aggregate materials to construct durable infrastructure is a priority. In general, the quantities of RMs are normally limited to 2 bags (~28 kg each) per material per request. International and out of Province requests for control and calibration materials are normally positively considered on a case by case basis. Where supplied, quantities are also normally limited to 2 bags per material per request.

Requests for large material quantities and/or requests intended for research purposes are also carefully considered by MTO on a case-by-case basis. In general, MTO requires additional information such as: impetus for the research; explanation of the research including the goals, scope, duration and potential benefits; if applicable, how the research supplements the work already completed using the same RMs requested; the quantity of material required and rationale for the quantity(s) requested. MTO reviews these requests in light of how the research may benefit the Province of Ontario and the general public interest. In addition, MTO reserves the right to limit quantities.

Where MTO chooses to provide materials for research purposes, it is done with agreement to the following conditions:

- Shipping is to be paid by the requestor. For materials shipped outside Canada, the requestor agrees to be responsible for, and to abide by, all importation rules and regulations specific to the country to which the aggregate(s) are being shipped, including names and contact information for customs/brokers; and
- Research completed using MTO RMs is published with acknowledgement of MTO’s contribution, i.e., a strong commitment is received from the requestor to share the outcome of the testing/research with MTO and the broader research community).

Requests for MTO RMs can be made by contacting soils-aggregates@ontario.ca. The requestor is asked to complete an MTO Reference Materials Request form. The form contains a listing of available RMs and their purpose, as well as detailed shipping instructions for the requestor. The completed form is sent to soils-aggregates@ontario.ca and MTO will respond accordingly.

Conclusions

The reference material program demonstrates MTO’s leadership and ongoing commitment to promoting quality testing and acceptance of soils and aggregates for more durable Ontario infrastructure. Various MTO RMs were established in the past, currently and are still being proposed for the future to support MTO’s mandate of being the leader in technical excellence. Through careful planning, sourcing, prospecting, acquiring, establishing and validating materials to date, over 20 RMs have been established and a few of them are still being maintained to support MTO Operations. As old RMs are depleted, new materials are being sourced and established to maintain the intended usage of the RMs including supporting other research and testing industries. The RM Code system has helped MTO categorize and classify the RM’s to their intended usage and facilitate management of RMs inventory.

MTO’s reference materials have also historically been used for and will continue to provide a solid foundation for test method development and research into the development of new and innovative technologies in the field of soil and aggregate engineering.

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